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भारतीय मानक

# पेट्रोलियम और उसके उत्पादकों की परीक्षण पद्धतियां

[पी : 55/वर्ग 2]

## तेल, वसा और मोम में साबुनीकरणीय और असाबुनीकरणीय पदार्थ का निर्धारण

(पहला पुनरीक्षण)

*Indian Standard*

# METHODS OF TEST FOR PETROLEUM AND ITS PRODUCTS

**[P : 55/Section 2]**

## SAPONIFIABLE AND UNSAPONIFIABLE MATTER IN OIL FAT AND WAXES

*(First Revision)*

ICS 75.080

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**BUREAU OF INDIAN STANDARDS**
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

*May* 2004

**Price Group 3**

Methods of Measurement and Test for Petroleum, Petroleum Products and Lubricants Sectional Committee, PCD 1

## FOREWORD

This Indian Standard [P : 55/Sec 2] (First Revision) was adopted by the Bureau of Indian Standards, after the draft finalized by the Methods of Measurement and Test for Petroleum, Petroleum Products and Lubricants Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

Saponifiable value, saponifiable and unsaponifiable matter, *see* [ P : 55] of IS 1448 was published in 1963 in order to align the method given in ASTM D 963 : 1954, which has since been withdrawn in 1963. It was decided to publish the separate test method, namely, Saponifiable value of petroleum products and saponifiable and unsaponifiable matter in oil fat and waxes.

The composition of the Committee responsible for formulation of this standard is given in Annex A .

In reporting the result of a test made in accordance with this standard, if the final value, observed or calculated, is to be rounded off it shall be done in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'

# *Indian Standard*

# METHODS OF TEST FOR PETROLEUM AND ITS PRODUCTS

## [P : 55/Section 2]

## SAPONIFIABLE AND UNSAPONIFIABLE MATTER IN OIL FAT AND WAXES

## *(First Revision)*

### 1 SCOPE

**1.1** This method [P : 55/Sec 2] is intended for determination of the saponifiable and unsaponifiable matter in blends of mineral and fatty oils. It is also suitable for essentially fatty materials.

**1.2** This method is not applicable, if the sample contains products saponifiable with difficulty such as certain waxes resins or esters of higher fatty acids which form sparingly soluble potassium salts. Also not applicable if sample contains highly polymerized fatty oils which forms emulsions that can be separated out only with difficulty.

### 2 SUMMARY OF METHOD

The weight sample is saponified with alkali and unsaponifiable matter is separated and weighed. The fatty acids are librated, separated and weighed.

### 3 TERMINOLOGY

**3.1 Saponification Value** — The number of milligrams of potassium hydroxide required to saponify one gram of the sample under conditions of this test.

**3.2 Unsaponifiable Matter** — The fraction of substances in fats or oils which are not saponified by caustic alkali but are soluble in ordinary fat-solvents.

### 4 APPARATUS

**4.1 Conical Flask** — 250/300 ml capacity alkali resistant glass and chemical clean.

**4.2 Reflux Condenser** — a water condenser.

**4.3 Hotplate/Water Bath**

### 5 REAGENTS

**5.1 Alcohol** — Absolute alcohol, 95 percent ethanol which has been purified in the following manner:

Dissolve 1.5 g of silver nitrate in about 3 ml of water add to 1 litre of alcohol in glass stoppered bottle and mix thoroughly. Dissolve 3 g of potassium hydroxide in 10 to 15 ml of warm alcohol, cool and add to alcoholic silver nitrate solution with slight stirring. Allow the precipitated silver oxide to settle, siphon of the clear supernatant solution and distill on a steam bath.

**5.2 Alcoholic Potassium Hydroxide 1 N Solution**— Dissolve 56 g of potassium hydroxide in 50 ml of distilled water and make up to 1 litre with alcohol.

**5.3 Dilute Hydrochloric Acid**, Approximately 3 N.

**5.4 Diethyl Ether**

**5.5 Methyl Orange Indicator**, 0.05 percent.

**5.6 Phenolphthalein Indicator**, 1 percent in alcohol.

**5.7 Anhydrous Sodium Sulphate,** Ignited.

**5.8 Potassium Hydroxide Solution,** 0.5 N approximately.

**5.9 Standard Sodium Hydroxide Solution,** 0.1 N.

**5.10 Acetone**

### 6 SAFETY PRECAUTIONS

Diethyl ether is a highly inflammable and its handling is carried out, away from flames or other sources of heat. All operations using diethyl ether must be carried out in fume cupboard.

### 7 PROCEDURE

**7.1** Use a quantity of sample such that not more than half of the potassium hydroxide added react during the test (*see* Note ). The following quantities are suggested :s

a) For oil containing more than 70 percent fatty matter use approximately 2 g of sample,
b) For oil containing 30 to 70 percent fatty matter use approximately 5 g of the sample, and
c) For oil containing less than 30 percent fatty matter use approximately 10 g of the sample.

NOTE — If larger amounts of unsaponifiable or saponifiable matter are required for further examination, larger samples may be taken provided the quantities of the reagents are suitably adjusted.

**7.2** Weigh the oil sample (to the nearest mg) into a saponification flask. Add 50 ml of 1 N alcoholic potassium hydroxide solution. Connect the flask to the reflux condenser. Boil the contents gently for 3 h, 1 h is sufficient for pure fatty oils. Before disconnecting the flask, wash down the condenser with 10 ml of alcohol. Cool the content of the flask and transfer these to chemically clean 500 ml separating funnel. Rinse the flask with 150 ml of water, followed by successive portions of ether to a total of 50 ml and all the rinsing to the separating funnel. Shake the mixture. During the ether extractions take care of release the pressure at frequent intervals. Allow the funnel to stand until the two layers separate and clarify. Draw off the lower aqueous alcoholic layer into the flask used for saponification.

**7.3** Drain the ether layer into second 500 ml funnel containing 20 ml water. If ether layer contains any solid suspended matter it shall be filtered into the funnel using a small, dry fat free filter. Wash the filter subsequently with small amount of ether and allow the washing to run into the main ether layer. Transfer the aqueous alcoholic layer back to the funnel and extract twice with more than 50 ml portion of ether. After each extraction run the aqueous alcoholic solution back into the reaction flask. Add each ether layer in turn to second funnel, filtering if necessary. Shake the content of second funnel gently, allow the layer to separate then run of the water into aqueous-alcoholic solution in the reaction flask.

### 7.4 Unsaponifiable Matter

**7.4.1** Add 2-3 ml of dilute hydrochloric acid to the content of second separating funnel and shake vigorously. Wash the ethereal solution with 20 ml of water and reject the washings.

**7.4.2** Wash the ethereal solution with a further 20 ml water and transfer these washings to the first separating funnel. Make three successive washings with 20 ml of aqueous potassium hydroxide solution then wash with 20 ml of portion of water until the washings are no longer alkaline to phenolphthalein indicator. Add all these washings to the first funnel.

**7.4.3** Transfer the ethereal solution from the second funnel to a chemically clean 600 ml beaker, rinse the funnel with a little ether and add to the beaker. Add 20 ml ether to the first funnel, shake gently and allow the phase to separate. Transfer the aqueous layer to the second funnel and wash the ether layer in the first funnel with successive 5 ml portion of water until the washings are no longer alkaline to phenolphthalein indicator. Add all the washings to the second funnel and transfer the ether layer to the main ether extract in the 600 ml beaker.

**7.4.4** Dry the ethereal solution in 600 ml beaker over anhydrous sodium sulphate until it is clear and bright transfer the solution through a fat free filter paper into a fared wide neck flask washing and sodium sulphate with a little dry ether. Distil off the ether on a steam bath. Heat the flask in an oven at a temperature of 80°C until it is substantially constant in weight and record the weight of the contents of the flask. Add 10 ml freshly boiled alcohol, neutral to phenolphthalein and swirl to dissolve the contents. Add a few drops of phenolphthalein indicator solution and titrate with 0.1 N sodium hydroxide to pink end point, the titration should not increase 0.1 ml. If it does reject the test and repeat from the beginning.

### 7.5 Saponifiable Matter

**7.5.1** Combine the aqueous-alcoholic solution in the reaction flask with the bulked washing from the second funnel in a 600 ml beaker and evaporate down to a convenient volume. Cool transfer to clean 500 ml separating funnel and acidify with an excess of dilute hydrochloric acid.

**7.5.2** Add 50 ml of ether to the contents of funnel, shake and allow the phases to separate. Draw off the acid layer into a beaker and pour the ether layer from the top of the funnel into a second funnel containing 20 ml of sodium sulphate solution. Repeat the ethereal extraction of the acid liquor twice more using in each case 50 ml of ether, combining the ether extracts in the second funnel. Shake the content of the second funnel allow them to separate and run off the sodium sulphate layer. Add a further 20 ml of sodium sulphate solution and repeat the procedure until the washings no longer give an acid reaction with methyl orange indicator.

**7.5.3** Dry the ethereal solution over anhydrous sodium sulphate until it is clear and bright, filtered into fared flask, washing the sodium sulphate with little dry ether. Remove the ether by distillation on steam bath when all ether apparently removed add 5 ml of acetone to the content of the flask and warm it on a water bath for approximately one minute. Remove the flask from the bath and then, while rotating the flask at an angle of about 45°, direct the gently current of air into the mouth of the flask for approximately one minute to remove the bulk of acetone.

**7.5.4** Place the flask in an explosion proof over at 80°C for 10 min. Remove the flask from oven, blow it with air as before for about 15 and allow the flask to cool in a desiccator for 10 min before weighing. Repeat this procedure until the difference between two consecutive weighings does not exceed 5 mg. Remove the last traces of ether by heating the flask in an oven at 100°C for not more than 30 min and then by blowing with a gentle current of air. Cool and weigh the flask. Record the weight of the contents of the flask.

NOTE — If it is known that fatty acids are very susceptible to oxidation, for example, instead fatty acids, drying in current of inert gas is an advisable.

## 8 CALCULATION AND REPORTING

Calculate the results as percentage of the sample and report them to the nearest 0.1 as the unsaponifiable matter and saponifiable matter.

## 9 PRECISION

### 9.1 Unsaponifiable Matter

Results of duplicate tests should not differ more than the following amounts:

Repeatability $\sqrt{0.13} \times (100 - X)$

Reproducibility $\sqrt{0.346} \times (100 - X)$

where $X$ is mean of duplicate results.

### 9.2 Saponifiable Matter

Results of duplicate tests shall not differ by more than the following amounts:

| *Fatty Acid Percent by Weight* | *Repeatability* | *Reproducibility* |
|---|---|---|
| Above 5 | 10 percent | 10 percent |
| Below 5 | Not established | Not established |

# ANNEX A

## (*Foreword*)

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*(Continued on page 5)*

(*Continued from page* 4)

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